

# TECHNICAL RESEARCH REPORT

## Characterization of Indentation impressions on Human Enamel for Hardness Measurement

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# **CHARACTERIZATION OF INDENTATIONIMPRESSIONS ON HUMAN ENAMEL FOR HARDNESS MEASUREMENT**

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## **ABSTRACT**

This paper presents results from investigating indentation impressions on human enamel under micro-hardness tests. The experiments of hardness testing were performed on a microhardness indentation machine under different loading conditions. Images of indentation impressions were obtained using an environmental scanning electron microscope. Geometrical shapes of hardness indentations were visualized in three-dimensional space using computer graphics. Quantitative information was obtained through atomic force measurements to characterize "pile-up", "sink-in", and elastic recovery of enamel. Special efforts have been made to study the microstructural effect of the calcified rods orientations on the fracture patterns formed during the hardness tests. Significant findings include that the occlusal surface demonstrates much stronger resistance to the indentation force than does the buccal surface and shows 40% elastic recovery after indentation. A new formula to determine hardness value has been proposed. By incorporating the reversible deformation into the evaluation, a normalized hardness measurement can be made to form a basis for comparison and other investigations where hardness has its unique role to play.

## **INTRODUCTION**

Advanced ceramic materials have been successfully developed during the past decades. They are widely used in a variety of applications for their superior properties, such as high-strength-to-mass ratio, excellent wear

resistance and exceptional corrosion resistance, compared to conventional materials, such as metals and plastics.

As new types of ceramic material emerge, assessment of their machinability becomes critical to ensure dental restorations made from them meet clinic requirements and patient needs. The research work presented in this paper is on microhardness tests of human enamel. The obtained information will be used as a critical reference to assess the feasibility of new ceramic materials for use as dental restorative materials. Behavior of material under the hardness testing in general reflects the characteristics of the material removal during machining. Therefore, results from the microhardness testing can be directly used to assess their machinability. It should be noted that the methodology employed in this research is new and different from those previously used in the dental research. It combines recently developed image processing technology, computer graphics, and fracture mechanics. Advanced measurement techniques, such as environmental scanning electron microscope and atomic force microscope, are used to provide quantitative information. The objective of this study is to investigate the behaviour of enamel material under hardness testing with emphasis on the effect of enamel rod orientation on the hardness. Such information for newly developed ceramic materials should be valuable assessment as it relates to material removal during shaping of dental restorations.

## SPECIMEN PREPARATION AND HARDNESS TESTING

Specimens prepared in the two different orientations, buccal and occlusal surface on the tooth, are tested on a Microhardness Indentation Tester Model 300, as illustrated in Figure 2. A Vicker's indenter is used. Eight loading conditions used vary from 50 grams to 2000 grams to model normal functional forces. Under each loading condition, two hardness indentations are performed. Consequently, sixteen indentation tests are done on a molar with a given orientation. Two molars with the identical orientation are used to duplicate the indentation tests. Therefore, a total of sixty-four indentation tests are performed on the exposed surfaces of the four molars in this study.

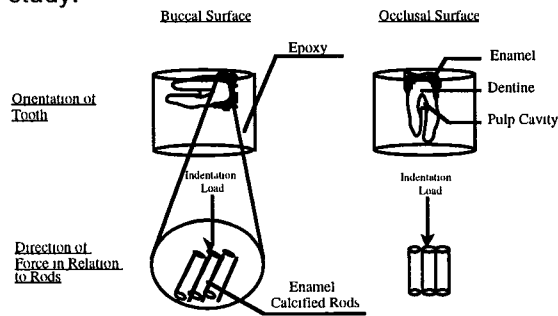


FIGURE 1. SPECIMEN PREPARATION OF THE HUMAN TEETH AT TWO DIFFERENT ORIENTATION OF THE CALCIFIED RODS

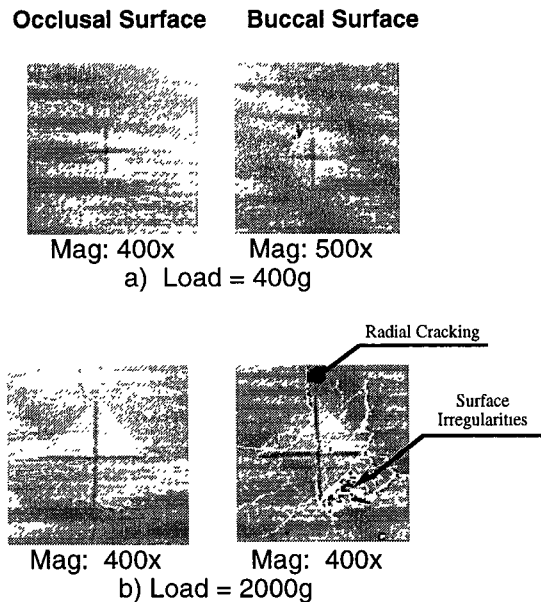


FIGURE 2. ESEM MICROGRAPHS OF INDENTATIONS ON THE OCCLUSAL AND BUCCAL SURFACE AT LOADS OF 400G AND 2000G.

Four surface impressions obtained from the indentation tests performed are illustrated in Figure 2. Among the four impressions, two are taken on the occlusal surfaces and two on the buccal surfaces. The loading conditions are 400g and 2000g, respectively.

To analyze the results obtained from the hardness measurements, the two curves illustrated in Figure 3 are constructed using Equation 1.

$$VHN = \frac{1.854 \cdot P}{d^2} \quad [\text{kg/mm}^2] \quad (1)$$

where: P = load, in kgf, and d = mean diagonal of indentation, in mm. Table 1 lists the mean and standard deviation of the hardness measurements.

The solid line represents the mean hardness values measured on the occlusal surface and the dashed line the mean hardness values measured on the buccal surface as the loading condition varies from the low to the high. It is evident that the measured hardness of the human enamel material, either on the buccal surface or on the occlusal surface, is a function of the loading condition. Examining the two plotted curves, a big hardness will be measured under a low loading condition, and a small value of hardness under a heavy loading condition. The variation range covers from 380 VHN [ $\text{kg/mm}^2$ ] when subjected to 400 grams force to 480 VHN [ $\text{kg/mm}^2$ ] when subjected to 50 grams force. Important findings from analyzing the results obtained from the hardness measurements are as follows:

(1) Both curves follow the same trend, i.e., the measured hardness value declines exponentially as the loading increases.

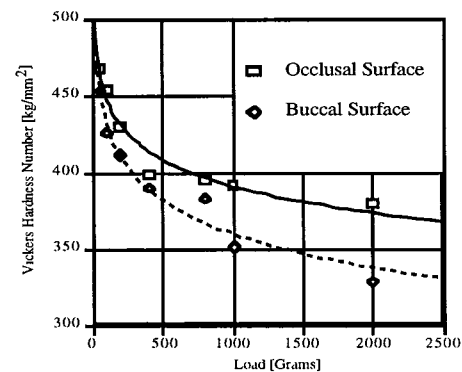


Figure 3. Hardness Curves of Enamel on Occlusal and Buccal Surface

(2)The occlusal hardness-load curve is consistently higher than the buccal hardness-load curve, indicating that the occlusal surface possesses stronger resistance to the indentation force than the buccal surface does.

### THREE-DIMENSIONAL TOPOGRAPHY ANALYSIS

In this study, we apply a new methodology called three-dimensional topography analysis. Through visualization of the hardness impressions, characterization of their geometric shapes is performed using methods of image processing and atomic force measurements. Figure 4 illustrates the developed computer-based system to carry out the characterization

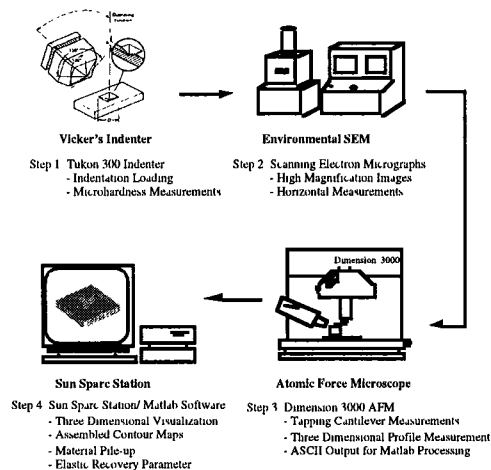


FIGURE 4.COMPUTER-BASED SYSTEM FOR CHARACTERIZATION OF MICROHARDNESS INDENTATIONS

### Atomic Force Microscopy

Elastic recovery at hardness indentations is a phenomenon well recognized by the research community, as illustrated in Figure 5.. Studies of impression geometries in metallic and non-metallic materials using various standard microhardness indenters indicate that whereas characteristic in-surface dimensions generally remains a reasonable measure of those at maximum loading, (thereby justifying a definition of hardness in terms of post-indentation measurements), the depth of the impression does not. Extremes in depth recovery are shown by 'soft' metals, where it is negligible, and 'highly elastic' rubbers, where it is nearly complete. Therefore, the phenomenon of elastic recovery is an important indicator characterizing the material property to resist the irreversible deformation.

Unfortunately, most of the formulas used for hardness evaluation, such as the formula presented in equation 1, do not count the factor of elastic recovery. As a result, an accurate assessment of material hardness is, if not impossible, difficult to achieve. This is especially true in evaluating the hardness of brittle solids where elastic recovery is usually significant.

In this study, a Dimension<sup>TM</sup> 3000 Atomic Force Scanning Probe Microscope is used to capture the topographic heights of the indentation geometry in micro-scale. The indentation topographic data are obtained using a sampling area of 64 x 64 nm and

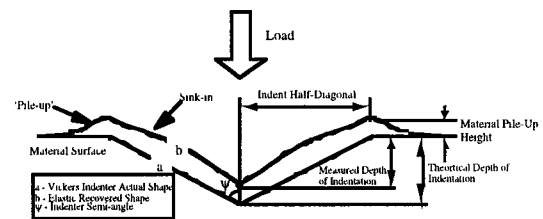


FIGURE 5. INDENTATION CYCLE AND ILLUSTRATION OF THE PROFILE OF INDENTATION

represents an image resolution of 256 x 256 pixels in both x and y direction. The Z direction accuracy is  $1 \times 10^{-3}$  mm and has a maximum depth measurement capability of 6 mm. In processing the data, several software tools are used, including NIH IMAGE and MATLAB.

### Three Dimensional Visualization of Indentation

3D image is reconstructed based on the Atomic Force Microscope measurements obtained from the indentations. Figure 6a presents two 3D isometric plots of the indentations at 200g and 400g on the occlusal surface, respectively. The two 3D isometric plots shown in Figure 6b illustrate the same loading condition on the buccal surface. The basic topographic features of the indentation, namely the 'pile-up', 'sink-in', and the shape of the diamond of the microhardness tester, are observed from these isometric plots. These features give a vivid picture of the deformation process during indentation. In the occlusal case, the rods are perpendicular to the applied load and do not fracture. Thus, a high hardness value is obtained for the occlusal surface and a small

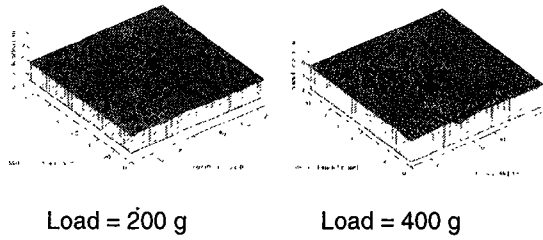


Figure 6a. Three Dimensional Topography Visualization of the Indents on the Occlusal Surface

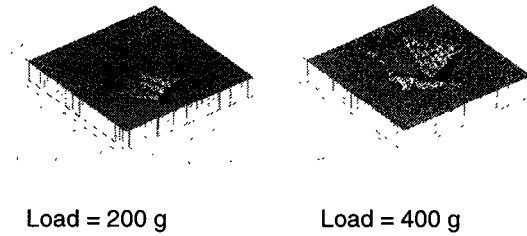


Figure 6b. Three Dimensional Topography Visualization of the Indents on the Buccal Surface

amount of pile-up and sink-in volume is found for both indentations at 200g and 400g, as illustrated in Figure 6a. In the buccal surface, the applied force acts at an angle to the rods, thus causing them to fracture and displace in various directions. The broken and misaligned rods appear in Figure 6b at the significant damage zone located on the edge of the impression, indicating possible shearing among the enamel rods due to its angular orientation nature on the buccal surface with respect to the loading condition. In addition, plastic flow of material forms 'pile-ups' and 'sink-ins'. The volume of these surface disturbances, as observed from Figure 6b, is relatively large if compared with the occlusal case.

## DISCUSSION OF RESULTS

Information gained from the 3D visualization offers great opportunities to understand the physical process of hardness indentation. The digitized data obtained from the atomic force microscopy measurements form a basis to perform quantification of the hardness indentation process. Two aspects discussed in this paper are elastic recovery of brittle material, and normalization of hardness measurements with respect to a selected loading condition.

### Elastic Recovery in Brittle Material

To quantify elastic recovery at hardness indentations, a new parameter is defined as follows:

$$\text{Elastic Recovered Index} = \frac{\text{Predicted Depth} - \text{Measured Depth}}{\text{Predicted Depth}} \times 100\% \quad (2)$$

In this study, the elastic recovery is calculated as a ratio of the difference between the predicted and measured depths to the predicted depth. It is expressed as a percentage. Note that an assumption is made in this study to calculate the predicted depth. It is assumed that the dimension of indentation diagonal remains unchanged during the elastic recovery process. Based on the assumption, the predicted depth is given by

$$\text{Predicted Depth} = (\text{Indent Half Diagonal}) \times \cot \psi \quad (3)$$

where parameter  $\psi$  represents the semi-angle of the indenter. It is equal to  $74.05^\circ$  in this study. It should note that the measured depth can be readily obtained by sorting the data in the depth direction and identifying the most negative number with respect to the reference planary surface, i.e.,

$$\text{Measured Depth} = \max \sum \text{abs}(\text{depth}_i) \quad (4)$$

Table 1 lists numerical values of the elastic recovery of hardness indentations. Data used for calculation are taken from the atomic force microscopy measurements for the two loading conditions of 200g and 400g for both occlusal and buccal surfaces.

Table 1. Results of the Indentation Height Parameters

Load (g)	Occlusal Surface		Buccal Surface	
	200g	400g	200g	400g
Measured Depth of Indentation( $\mu\text{m}$ )	2.59 $\mu\text{m}$	3.20 $\mu\text{m}$	2.86 $\mu\text{m}$	3.24 $\mu\text{m}$
Theoretical Depth of Indentation( $\mu\text{m}$ )	4.21 $\mu\text{m}$	6.23 $\mu\text{m}$	4.21 $\mu\text{m}$	6.23 $\mu\text{m}$
Elastic Recovered Index (%)	37%	49%	32%	48%

Examining the listed data, two important observations can be made. The first observation is that elastic recovery along the depth direction is significant at low loading conditions, such as 37% at 200g versus 49% at 400g for the occlusal surface. In fact this is true for both occlusal and buccal surfaces. The second observation is that the elastic recovery is more significant with the occlusal surface than the elastic recovery with the buccal surface, such as 37% versus 32% at

200g, and 49% versus 48% at 400g. However, such differences decrease as the loading condition increases. This is due to the presence of a dominant crack system usually formed under high loading conditions.

### **Normalization of Hardness Measurements**

The hardness values measured under different loading conditions differ from each other significantly. As shown in the data listed in Table 1, the maximum number is 468 VHN at 50g and the minimum number 379 at 2000g for the occlusal surface. Therefore, telling a hardness number without giving the corresponding loading condition may not provide sufficient information on the hardness property of the material. Normalization of the hardness measurements made under different loading conditions with respect to a selected, or commonly acceptable, loading condition is needed.

If examining the formula used to determine the hardness value from measurement, such as Equation 1, a surprising fact is that the geometrical parameter used is the dimension of the impression diagonal. This parameter has the least effectiveness in characterizing the reversible deformation occurred during indentation. The credibility of determining hardness value without taking to account the reversible deformation, such as elastic recovery of the indentation depth, is in question.

In this study, a procedure to perform normalization and incorporate the reversible deformation into the hardness determination is proposed. Equation 1 is revised and two new parameters are introduced.

$$\text{Normalized Hardness} = \frac{1854 * P}{d^2} * \sqrt[4]{\text{Elastic Recovered Index} * C} \quad (5)$$

The first parameter is based on the elastis recovery index. Recognizing the non-linearity between the material hardness and elastic recovery, a fourth root is used. As an example

$$\text{Normalized Hardness} = \frac{1854 * P}{d^2} * \sqrt[4]{\text{Elastic Recovered Index}} \quad (6)$$

before normalization:	468	453	430	398
elastic recovery index	29%	25%	37%	49%
after normalization:	328	331	334	326

to demonstrate the introduction of this parameter, the following data are before and

after the normalization for the occlusal surface together with the four numerical values of elastic recovery index:

As indicated, the normalized hardness values are 328, 331, 333, and 326, that are very close to each other and vary slightly about 330 HVN. Numerical values of these four normalized hardness measurements may not seem reasonable if comparing their values measured before normalization. These values are 468, 453, 430, and 398, respectively. Therefore, there is a need to make an adjustment for the normalized hardness value be set at a given loading condition. Say the loading condition of 200g is selected. The parameter C in equation 8 can be set at the following value:

$$C = \sqrt[4]{3} = 1.316 \quad (7)$$

Using this correction factor to adjust the normalized hardness values will allow us to raise the average of the normalized hardness values to a new level. It is now at 430 HVN because the four normalized hardness values after adjustment are 431, 435, 439, and 429 HVN, respectively, as illustrated in Figure 8. Graphically speaking, such a normalization and adjustment process is to bend a curve to a horizontal line and then shift the line upward or downward. In this study, the shift value is 100 VHN.

The significance of implication of this normalization process is two-fold. First, the normalized hardness values are determined not only by the applied load and the measured diagonals of the impression, but also by incorporating the reversible deformation into the hardness evaluation. As a result, the normalized hardness value will have a unique hardness value for a given type of ceramic material without specifying the loading condition under which a specific indentation test is performed.

The second contribution of this normalization process is the establishment of a criterion to use the hardness measurement as a tool for comparison among different ceramic materials. In this study, we obtained two curves, one for the occlusal surface and the other for the buccal surface. Using equation 5 and having parameter C set at 1.316, the normalized hardness value for the occlusal surface is 435 the normalized hardness for the buccal

surface is 420 VHN. Therefore, comparison b

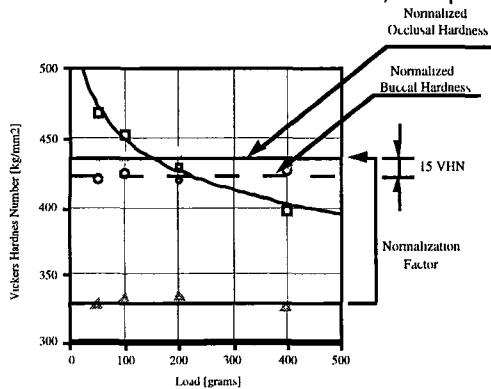


Figure 7. Illustration of Hardness Normalization

etween VHN, and the two normalized values clearly indicates that the hardness of the occlusal surface is higher than that of the buccal surface. The difference is about 6-10 %, mainly due to the orientation of rods with respect to the loading surface.

## CONCLUSIONS

Significant findings are summarized as follows.

1. Vickers Microhardness indentation using loading conditions, from 50g to 2000g has been performed on both occlusal and buccal surfaces in human enamel. Significant cracking fracture and deformation patterns have been observed.
2. A computer-based Scanning Electron Microscope and Atomic Force Measurement Analysis system has been used to perform surface height measurements of the indentation topography. These measurements are used to visualize hardness indentations using computer graphics software. Three types of visualization plots of the indentation have been presented, 1) isometric plot, 2) contour map, and 3) inverted isometric plot, providing a vivid picture and rich information into the material fracture and deformation during the indentation process.
3. A new formula to determine microhardness values for indentation tests is proposed in this research. By incorporating elastic recovery into evaluation, a normalized hardness measurement can be achieved to associate a given type of ceramic material with a unique hardness value. This approach offers a unique opportunity to use hardness measurement as a means for comparison and other

investigations related to material hardness characteristics.

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